MODELLING OF THE DIP-COATING PROCESS

Dipcoating, wherein an object is withdrawn from a liquid bath, retaining a thin layer of liquid which then dries, is perhaps the simplest of coating techniques. SOLA Optical asked the MISG to investigate this process as a means of applying a protective coating to spectacle lenses. The theory, as proposed by the group, involves the 'lubrication' form of the equations of slow viscous flow. This yields an estimate of the wet coating thickness variation, which in turn depends on the liquid properties, the pull rate, and the lens curvature. When this thickness is reduced by the volatile fraction of the coating, predicted thicknesses are in rough agreement with reported values. Once applied, the liquid film drains downward and this effect is modelled numerically. An outline of a more complete model, allowing evaporation and drying, is also presented.

1. Introduction

The SOLA Optical Company supplied the group with data obtained for a variety of lenses, pulled at various speeds, and using two different coating compounds. Typically lenses to be coated varied between 65 and 75 mm in diameter and had at least one curved face. The lenses were pulled vertically upward, out of the coating bath, at speeds U between 10 and 50 cm per minute. A simplified schematic drawing is shown in figure 1. The nonevaporating component, termed 'solids', constituted between 10 and 20 per cent of the initial coating liquid formulation. While the final dry coating has significant nonuniformities in thickness, a single coating thickness is reported for each lens. This may be interpreted as an average value.

The dipcoating process has important advantages for spectacle lenses if it can be made to produce satisfactory performance. It may replace spin coating, in which a quantity of liquid is placed upon a lens blank and then spun at high speed; the excess is thrown off and a uniform final thickness results. However spin coating is inherently more involved and also requires each lens to be treated individually.

The mathematical description of the dipcoating process involves the flow of a viscous liquid with a free surface. Surface tension acts on the surface and is important in determining the coating thickness. Until sufficient volatiles have evaporated, the drawn liquid layer remains mobile and drains downward under the action of gravity. A complete description of the process must also include a

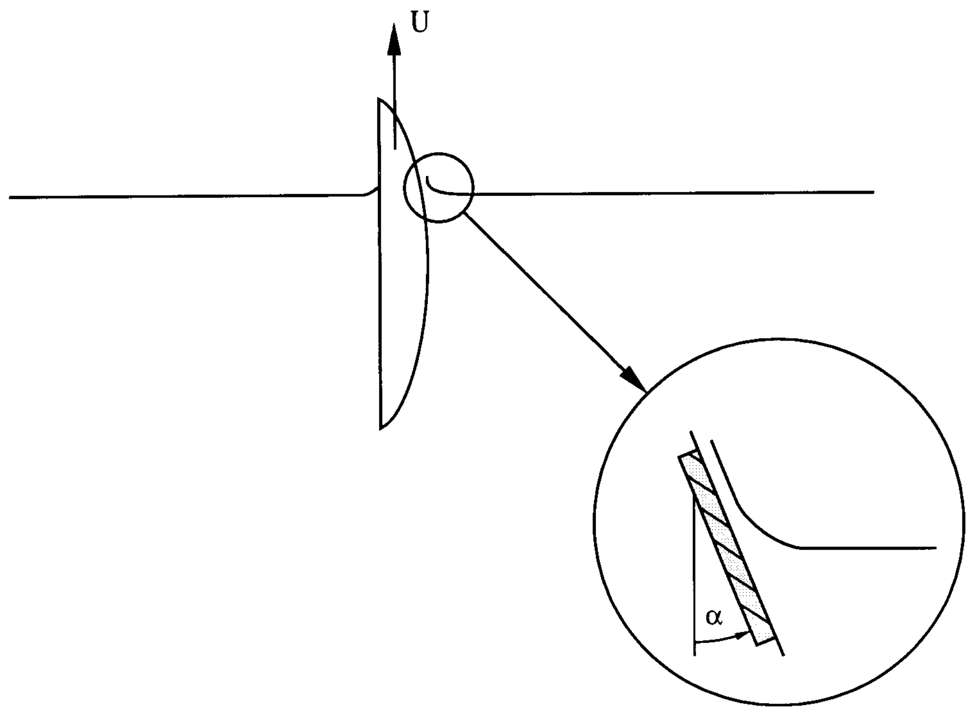

Figure 1: A lens being drawn upward from a liquid bath, showing a magnified view on the convex face.

model for drying with consequent loss of coating volume and large increase in viscosity. In addition, it may be that certain coatings have complicated rheological behavior and their effective viscosity may depend on the local details of their motion in addition to the local value of the solids fraction. Preliminary estimation for this process, as developed below, will assume a relatively simple model wherein only the unsteady flow of a Newtonian, nonevaporating liquid will be considered.

Steady-state vertical withdrawal of a moving surface or 'substrate' from a Newtonian liquid bath was first treated by Landau and Levich (1942). Their theory gives good estimates of the coating thickness when the capillary number, defined as the product of fluid viscosity times drawing speed, divided by the surface tension, is a sufficiently small number, as it is in the present application. In the following section, the basic components of this theory will be outlined, along with modifications necessary to treat the varying inclination of the lens elements when a curved lens is withdrawn from the bath. This may be thought of as the original coating distribution. The drainage of this initial distribution is treated in section 3 via a numerical solution of an approximate unsteady equation. It will be seen that drainage will result in a substantial redistribution of the coating in times of the order of one minute. Both two and three dimensional simulation results will be shown; the simpler two-dimensional case appears to capture the important features except for some transverse, or 'sideways' flow along the lower edge of the lens.

A more complicated theory, allowing for evaporation and drying, will be presented in section 4. While no numerical results will be given here, this more complete theory has been used with significant success in related problems. The concluding section will discuss the status of the modelling effect relative to the known experimental results. The need for further experimental measurements will be highlighted.

2. A local model for the prediction of coating distribution

We consider the steady-state flow of Newtonian or almost-Newtonian fluids in a thin layer of height h with a stress-free surface. With the additional assumptions that the motion is slow, and the free-surface is almost parallel to the substrate, the governing momentum equation may be approximated as

$$- p_x + \mu u_{yy} = 0. \tag{1}$$

Here subscripts signify partial differentiation, μ is viscosity, p is pressure, u is the velocity parallel to the substrate, x and y are coordinates measured parallel and normal to the substrate respectively. Equation (1) is the basis of the lubrication theory and its asymptotic validity has been well established. (See e.g. Sherman, 1990). Because of the thinness of the liquid layer, p depends only on x and t, and, assuming μ is also independent of y, (1) can be integrated immediately to give a parabolic velocity profile in y. The pressure gradient within the thin layer is due to changes in the curvature of the free surface, upon which surface tension σ acts. Since the slope of the free surface is assumed small, the surface curvature is approximately h_{xx} and the pressure gradient within the layer is σh_{xxx}. If the pressure-driven flow is exactly balanced by the backwards motion of the wall, i.e. wall motion in the negative x direction with speed U, it is possible to have a steady-state free surface. The flux Q within the layer is given by

$$Q = \int_0^h u \, dx = \frac{\sigma h^3 h_{xxx}}{3\mu} - Uh = -Uh_\infty \tag{2}$$

where h_∞ is a constant or reference layer thickness. Equation (2) is an ordinary differential equation for steady-state coating flow with a free surface. All physical constants can be absorbed in the scaling

$$h = h_\infty H, \qquad x = \left(\frac{\sigma}{3\mu U}\right)^{1/3} h_\infty \xi \tag{3}$$

resulting in the so-called Landau-Levich equation

$$H^3 H_{\xi\xi\xi} = H - 1. \tag{4}$$

Equation (4) can be used to represent the shape of the coating film on an object that is being continuously drawn from a liquid bath. A discussion of the mathematical properties of this, and closely-related equations can be found in the recent survey paper of Tuck and Schwartz (1990). If, starting from an almost uniform film, the equation is integrated in the positive ξ direction, it will ultimately yield a constant second derivative $H_{\xi\xi}$ as ξ goes to ∞. This limiting value of the second derivative, determined by numerical integration, is about 0.643 . Because the limiting value is obtained while the slope remains sufficiently small, the second derivative may be identified with the limiting value of the surface curvature $1/R$. Returning to the original variables, using equation (3), one obtains, as in Bretherton (1961),

$$h_\infty / R = 0.643 \left(\frac{3\mu U}{\sigma} \right)^{2/3}. \tag{5}$$

R may be interpreted as the radius of curvature of the liquid surface where the drawn film meets the bath. This 'meniscus' shape is determined solely by a balance of capillary and gravity pressures and appears, since the drawn film is thin, identical to the static meniscus on a straight wall. The radius of curvature of the static meniscus, for a vertical wall, is

$$R = \left(\frac{\sigma}{2\rho g} \right)^{1/2}.$$

Combining this expression for R with equation (5) gives an estimate for the constant film thickness in steady vertical drawing from a liquid bath:

$$h_\infty = 0.946\, (\mu U)^{2/3}\, \sigma^{-1/6}\, (\rho g)^{-1/2}. \tag{6}$$

Apart from an erroneous value for the numerical constant in equation (6), this is the result given by Landau and Levich. Some data on film thicknesses has been provided by SOLA. They give the drawing speed U, viscosity μ, and solids fraction c_0 as well as measured dry coating thicknesses for each case. A simple theoretical prediction, that may be compared with the data, is based on the assumption that the wet film thickness is as given by equation (6), and that no further flow takes place. The dry film thickness is the wet thickness multiplied by the supplied value of c_0. No details of the lens shape are included in such a model; nor is it clear, in either the data or the simple theory, at which points, on a nonplanar lens, the stated thicknesses are to be found. Figure 2 compares predictions of dry thickness versus drawing speed with reported measurements for two different coating formulations. While the simple theory appears to correctly predict the magnitude of the dry thicknesses and gives reasonable agreement for low drawing speeds, it overpredicts the measured thicknesses at the highest speeds by a factor of almost two. Formulation A is a liquid of 9.5 Cp viscosity and a solids fraction c_0 of .225 while, for Formulation B, the corresponding figures are 5.6 Cp and .133 . Certain of the relevant physical variables in equation (6) were not reported by SOLA. We have assumed the following typical values for organic liquids: $\sigma = 30$ dynes/cm and specific weight $\rho g = 900$ dynes/cm^3. The error brackets on the data, 0.3 μm, are as given by SOLA. Finally, it should be mentioned that only two of the four supplied data sets are shown in figure 2. The ones not shown are both for Formulation A; one set was quite similar to results shown for this formulation, while the other appears anomalous in that it gives thicknesses that uniformly exceed the theoretical prediction.

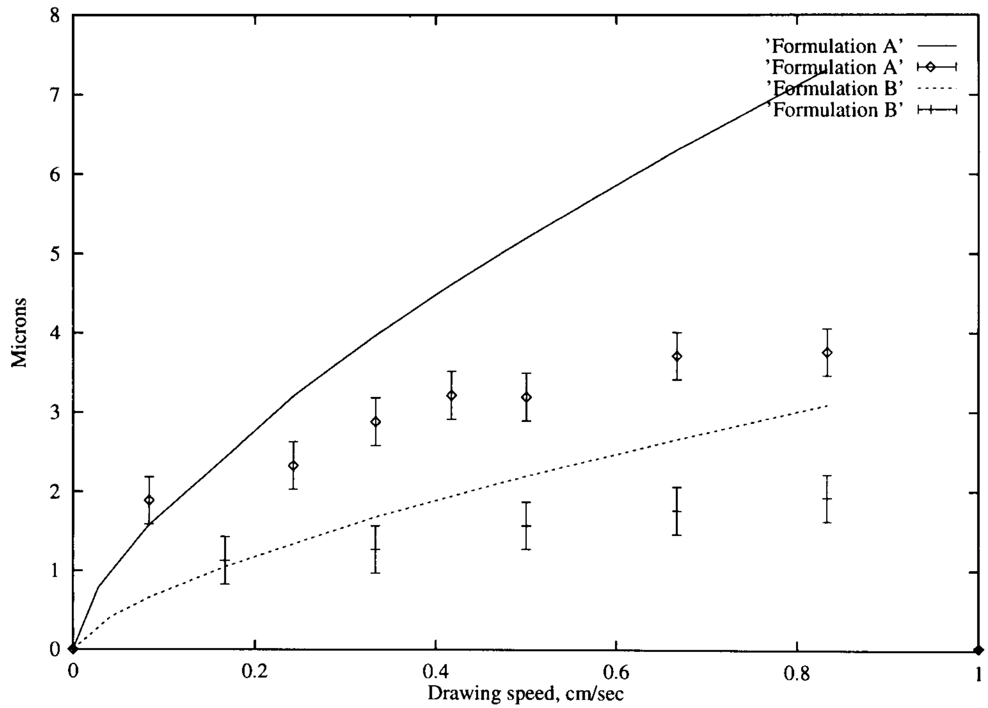

Figure 2: Dry thicknesses for 2 formulations; simple theory versus experiment.

The validity of the formula in (6) has been established, by Levich (1962) and others, for continuous drawing processes, by comparison with experimentally measured values, for values of capillary number $(\mu U/\sigma) < O\ (10^{-2})$. The data in figure 2 satisfy this requirement. It is likely, therefore, that thinning of coating, due to gravity drainage, is important for the larger values of original thickness. This will be discussed further in section 3. For curved lenses, an additional requirement concerning the smallness of the film-formation time, must also be met, as discussed below.

2.1 Effect of lens curvature

Candidate lenses all have one convex surface, with the reverse side either concave or flat. The local model given above can easily be extended to curved surfaces. Thus a spherical convex surface, when pulled vertically from a bath, initially has a 'meniscus turning angle,' i.e. the angle between the surface tangent plane and the horizontal bath where they meet, that is greater than $\pi/2$. This angle diminishes continuously as the lens is withdrawn, and is less than a right angle for the 'lower' half of the lens. If α is taken as the angle between a surface tangent and a vertically-upward vector, a 'local' theory assumes that the laid-down wet coating will be the same as that for a straight plate being drawn out of the bath at angle α.

The radius of curvature of the meniscus, required in equation (5), now depends on α and the static calculation needs to be modified. Consider the liquid surface extending from the 'contact' point on the plate, where its downward inclination angle is $\theta_0 = \pi/2 - \alpha$, to the horizontal bath which may be taken as $z = 0$. Letting θ be the general inclination of the free surface from the horizontal, the balance of pressures at the surface is given by

$$\sigma \left(\frac{d\theta}{ds}\right) = \sigma \left(\frac{d\theta}{dz}\right) \left(\frac{dz}{ds}\right) = \rho g z. \tag{7}$$

Now $dz/ds = \sin\theta$ and (7) is a first order differential equation for $\theta(z)$ that can be integrated immediately. Far away from the wall the liquid surface is flat, giving the boundary condition $\theta = 0$ at $y = 0$. The radius of curvature at the contact point can now be found from the elevation there, using (7), as

$$R(\alpha) = \left(\frac{\sigma}{2\rho g}\right)^{1/2} (1 - \sin\alpha)^{-1/2}.$$

For a lens drawn vertically upward at speed U, the effective drawing speed for an inclined element is $U/\cos\alpha$ and this modification also needs to be introduced into equation (6). The modified α-dependent initial coating thickness is

$$h_\infty(\alpha) = 0.946\,(\mu U)^{2/3}\,\sigma^{-1/6}\,(\rho g)^{-1/2}\,(\cos\alpha)^{-2/3}\,(1 - \sin\alpha)^{-1/2}. \tag{8}$$

The steady-state film thickness on an inclined plate appears to have been first treated by Wilson (1982).

Two further points should be made concerning the local applicability of equation (8). For a spherical cap lens drawn upward, the local angle α is constant on any horizontal level. Thus the longitudinal curvature does not introduce any further complication, and the wet film thickness is invariant horizontally. Secondly, the local use of (8), an infinite plane result, requires some justification. Fundamentally this requires that α have negligible change in the distance required to 'set up' the film. From the scaling in (3) and the result (6), this distance is $O(Ca^{1/3}L_c)$ where L_c is the 'capillary length' $\sqrt{\sigma/(\rho g)}$. The fractional change in thickness over this scale $\Delta h_\infty/h_\infty$ can be shown to be $O(Ca^{1/3}L_c/\rho_0)$ where ρ_0 is the radius of curvature of the lens. In the present application, a typical value for this parameter is 10^{-4}.

3. Film drainage

Until such time as the volatile component has largely evaporated, the applied film will drain downward under the action of gravity. The profile shape will change with time. Calculations, for this process, can be made by use of the two-dimensional model equation

$$3\mu h_t = -3\mu Q_s = -\left(\rho g h^3 + \sigma h^3 h_{sss}\right)_s \tag{9}$$

subject to the initial condition that $h(s,0)$ is given, and the boundary conditions are that the flux is zero at the edges,

$$Q(0,t) = Q(L,t) = 0$$

where s is arc length and $s = 0, L$ are the upper and lower ends of the lens respectively. For a plane vertical lens, according to the simple theory of the last section, $h(s,0)$ can be equated to the constant value h_∞ given by equation (5) above. If lens curvature is considered, then $h(s,0)$ can be taken as $h_\infty[\alpha(s)]$ from equation (8); here, the specified function, giving inclination angle α in terms of arc length, is particularly simple for a circular-arc lens of radius ρ_0 where

$$\alpha = \frac{L}{2\rho_0}\left(1 - \frac{2s}{L}\right).$$

Equation (9) is made dimensionless, with parameters absorbed in the scaling by taking

$$h = h_0\hat{h}, \qquad s = \left(\frac{\sigma h_0}{\rho g}\right)^{1/3}\hat{s} = L_c^{2/3}h_0^{1/3}\hat{s},$$

$$t = \left(\frac{3\mu L_c^{2/3}}{\rho g h_0^{5/3}}\right)\hat{t}.$$

Here h_0 is the drawn wet film height on a vertical plate, which can be written as

$$\frac{h_0}{L_c} = 0.946Ca^{2/3}.$$

The dimensionless equation is

$$\hat{h}_{\hat{t}} = -\left[\hat{h}^3\left(1+\hat{h}_{\hat{s}\hat{s}\hat{s}}\right)\right]_{\hat{s}} \tag{10}$$

where, for a curved lens,

$$\hat{h}(\hat{s},0) = (\cos\alpha)^{-2/3}(1-\sin\alpha)^{-1/2}.$$

Equation (10) has been solved numerically using a finite-difference scheme. Time integration is done implicitly to ensure stability. Details of a similar scheme can be found in Moriarty *et al.* (1991). For a two-dimensional convex lens profile (L = 70mm, ρ_0 = 100mm) and parameter values corresponding to Formulation A, drainage histories for two draw speeds, U = 100 mm/min and 500 mm/min, are shown in figures 3 and 4. Profiles are shown, for each case, at 0, 30, and 60 seconds. For the low speed case, the profile over most of the lens changes little from the prediction of equation (8). The general tendency remains for the coating to be thicker at the top (left side in the figure) of the lens. A thick edge forms at the lower boundary, reaching a height of about $3h_0$ at t = 60 sec. At the middle of the lens, the thickness is only a little greater than predicted by the simple theory. If one now were to assume that flow terminates after 60 seconds, at which time the coating dries, the rather close agreement between theory and experiment in figure 2, for low speeds, can be understood.

The situation is quite different for the higher speed case, where the drawn film is much thicker, shown in figure 4. There after 60 seconds, a much greater fractional drainage has occurred. The height of the thick lower edge is about 16 times h_0. The general tendency is for the film to be thicker at the bottom and, at the center, after 60 seconds, the local thickness is about $0.8h_0$. This provides at least a qualitative understanding for the overprediction of the simple theory in the higher-speed cases in figure 2.

For both cases shown, the evolution of the profile in the neighborhood of the top does not depend on capillary forces. The shape variation there is in accordance with the prediction of Jeffreys (1930) who used the method of characteristics to solve the first-order partial differential equation that results when the surface tension term in (9) is neglected.

While gravity drainage seems to explain qualitative features of the experimental results, certain comments need to be made concerning the quantitative correctness of the above numerical predictions. (i) The nominal value of 60 sec for flow, followed by drying in place, is somewhat arbitrary; it does, however, reflect the approximate interval before polymerization that was reported to the Group. The assumption that the viscosity remains constant during the flow period is a gross approximation. This will be discussed further in the next section.

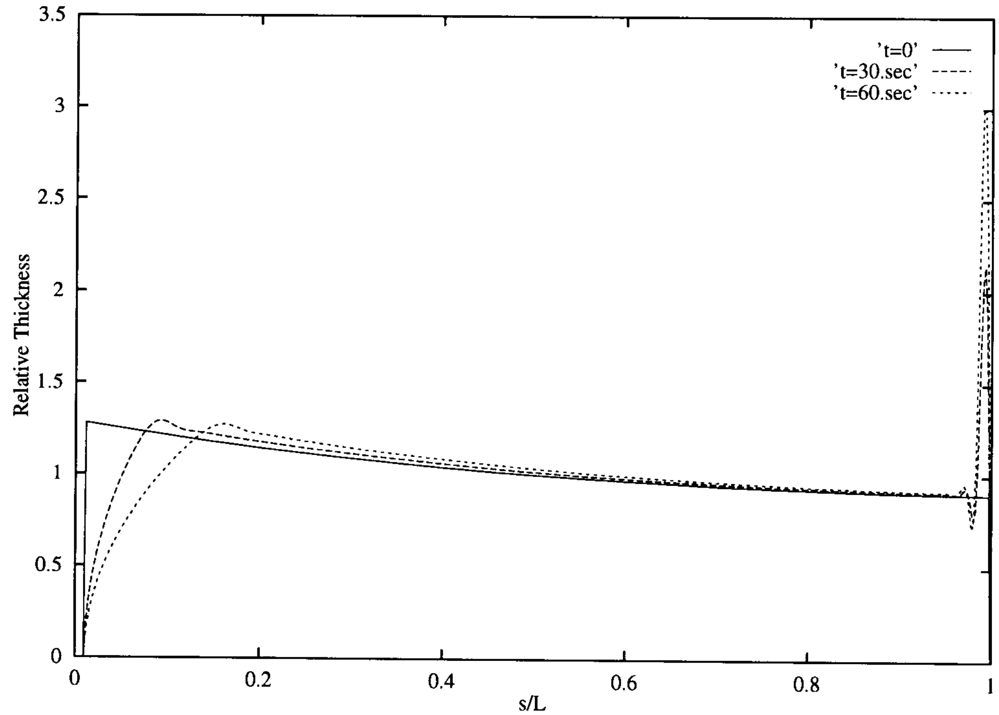

Figure 3: Draining profiles, low speed draw; U = 100 mm/min.

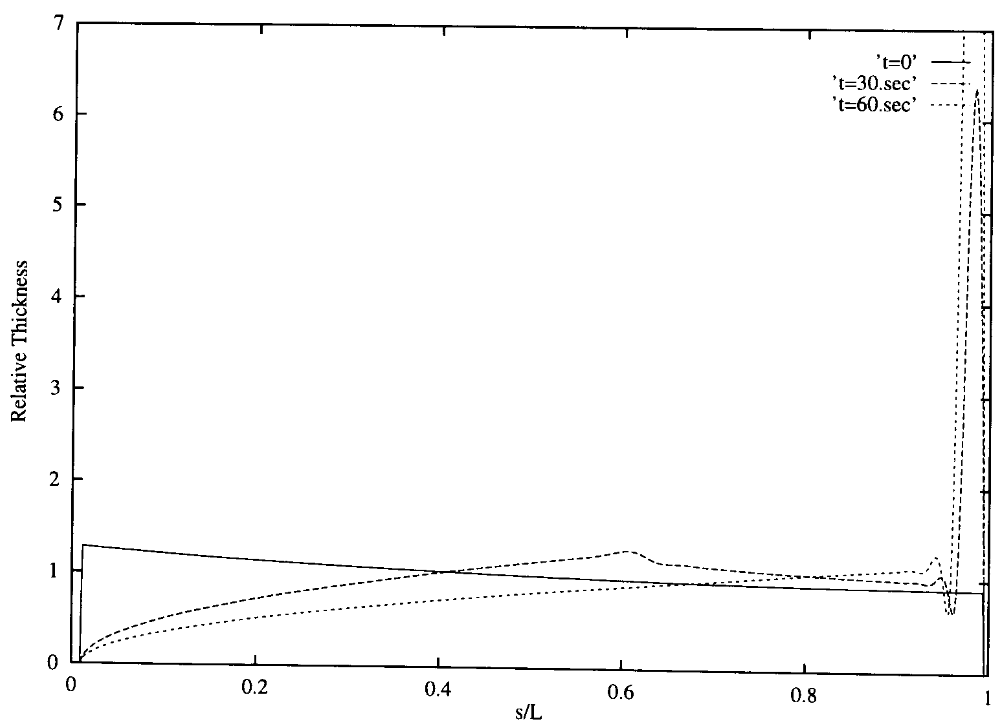

Figure 4: Draining profiles, high speed draw; U = 500 mm/min.

(ii) A more correct lubrication equation, for a curved lens surface, replacing (9) is

$$3\mu h_t = -\left(\rho g h^3 \cos\alpha - \rho g h^3 h_s \sin\alpha + \sigma h^3 h_{sss}\right)_s \tag{11}$$

reflecting the local drainage behaviour on an inclined lens element. Thus the tangential component of gravity is reduced by the factor cos α and a normal component of gravity also appears. This latter component can tend to either level or 'de-level' the film, according to whether α is greater than or less than zero. In particular, it may cause film instability on the lower half of a convex lens. To ascertain the importance of these modifications, a case was run using the improved equation (11). For a lens whose radius of curvature is 100 mm, the maximum inclination, either positive or negative, is about 20 degrees from the vertical. The differences between the use of the two equations were rather small, and, unless lenses of significantly greater curvature need to be considered, the more complex equation is not warranted. (iii) It has been assumed, in the simulations, that the lens is completely coated during drawing before downward draining is allowed. This is potentially a serious limitation, but is one that could be corrected in a more detailed model. Clearly downward drainage commences immediately after withdrawal of each lens element. At low speeds, e.g. 100 mm/min as in figure 3, the withdrawal time for a 70 mm diameter lens is 42 sec. This is a significant fraction of the 60 sec nominal drainage time. However, apart from the height of the 'fat edge' on the bottom, a large change is not expected since relatively little flow occurs. For the high speed case, omission of immediate drainage is perhaps more serious; although, at 500 mm/min, the withdrawal time is only about 8 seconds.

At somewhat greater computational cost, it is possible to calculate three-dimensional drainage. Such an unsteady calculation is desirable in order to determine the effect of horizontal flow components. An alternating-direction, partially-implicit finite difference scheme has been developed for these problems. It is similar in principle to the ADI method, developed for the linear diffusion equation as discussed by Peaceman and Rachford (1955). The partial differential equation, replacing (10) is

$$\hat{h}_{\hat{t}} = -\nabla.\left[\hat{h}^3\left(1 + \nabla\nabla^2\hat{h}\right)\right] \tag{12}$$

where ∇ is the two-dimensional operator $(\partial/\partial x, \partial/\partial y)$ and (x, y) is an othogonal coordinate system on the surface of an almost planar lens.

Because a rather fine mesh is needed to resolve the developing fat edge at the lens bottom, a smaller-diameter lens was used as a test case. The (x, y) mesh employed had the dimensions 180 x 90 and the lens was taken to be planar. A no-flux condition was enforced on the circular boundary. Results are shown in figures (5a) and (5b) for dimensionless time $\hat{t} = 3.4$. In (5a), for reference purposes, the calculated two-dimensional profile is shown. Note that the maximum height of the fat edge is $2.63h_0$. In the three-dimensional case, in figure (5b), where only one half of a bilaterally-symmetric profile is shown, the maximum lip or edge thickness, on the centerline, is calculated to be $3.67h_0$, the greater height resulting from side flow along the lower edge of the lens. The results shown in figure (5b) include both a perspective view of the free surface, as well as a set of equi-height contours. They show some similarity to the steady-state, or final, solutions calculated by Tuck and Schwartz (1991). Because of the nondimensionalization used, the results of figures (5) pertain to a family of physical situations. For drawing speed $U = 100$ mm/min and the lens diameter $L = 12.0$ mm, the elapsed time is 29 sec, while for $U = 500$ mm/min and $L = 17.3$ mm, the elapsed time is 4.9 sec. No difficulty, apart from a significant increase in computational load, is anticipated in the three-dimensional calculation for 'full-size' lenses.

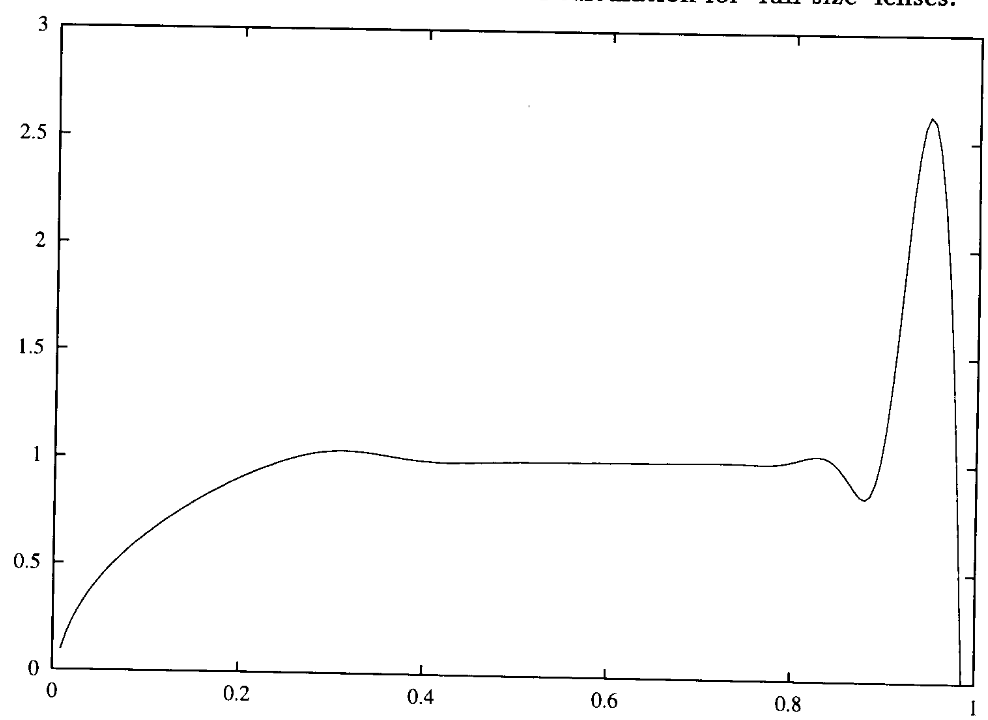

Figure 5(a): Two-dimensional draining, small lens, $\hat{t} = 3.4$.

4. Outline of a more complete theory

The assumption that the viscosity of the liquid coating remains constant is a serious limitation for highly volatile liquids, such as used in the present application. Typically, viscosity is a strong function of component fractions for a complex liquid and a significant increase in viscosity could occur on a time scale of one minute or less. The simplest theory that may capture this effect involves the use of a two-component model.

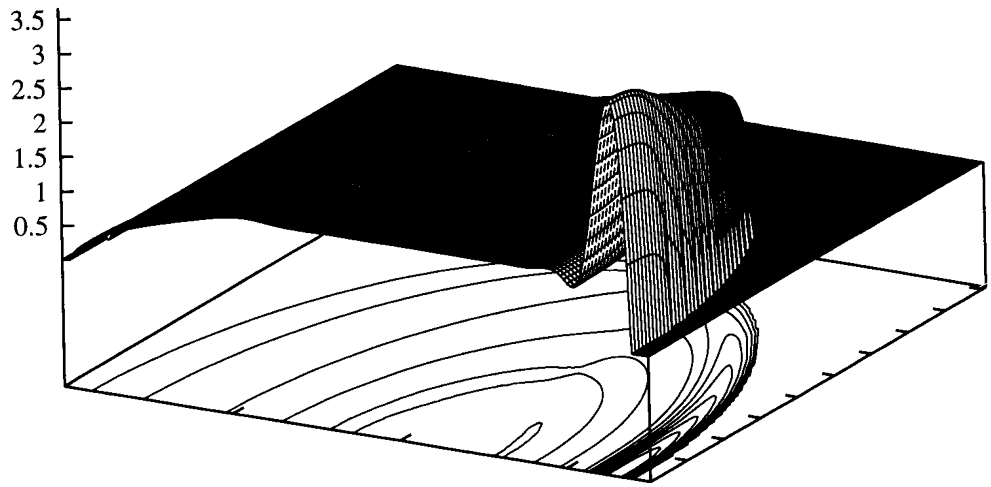

Figure 5(b): Three-dimensional draining, small lens, $\hat{t} = 3.4$.

The liquid is taken to consist of two components termed "resin"(or "solids") and "solvent"; only the solvent component is assumed to be volatile. The resin fraction, or concentration c, is taken to be uniform across the thin film. The validity of this "well-mixed" assumption rests upon certain assumptions concerning species diffusion, evaporation rate, and pressure gradients. Let E be the evaporation rate, with dimensions of thickness per unit time. E will be a function of the local value of c, in general. If $T_{\text{dry}} \sim h/E$ is a characteristic time for drying and $T_{\text{diff}} \sim h^2/D^{(r)}$ is the characteristic diffusion time, where $D^{(r)}$ is the diffusion coefficient for resin in the bulk liquid, uniformity of c across the layer requires that

$$T_{\text{diff}} \ll T_{\text{dry}}$$

or

$$h \ll D^{(r)}/E. \tag{13}$$

This criterion can be satisfied for sufficiently thin layers.

The concentration changes due to evaporation, convection, and diffusion along the film according to

$$c_t = \left(\frac{E}{h}\right) c - \left(\frac{Q}{h}\right) c_s + D^{(r)} c_{ss}. \tag{14}$$

The evaporation rate can be modeled as a power-law function of concentration

$$E(s,t) = E_0(1 - c(s,t))^n \tag{15}$$

where n is an experimentally determined constant ($0 \leq n \leq 1$) and E_0 is a constant with units of velocity. Equations (14) and (15) incorporate the basic result that regions of thin coating dry faster than regions of thick coating.

The viscosity is taken to depend on concentration; a suitable law, after Patton (1979) is

$$\frac{\mu}{\mu_0} = A\exp(Bc) \tag{16}$$

where A and B are empirically derived constants and μ_0 is the initial viscosity. The evolution equation for the surface shape must be modified by inclusion of an evaporation term and the viscosity is now variable in space and time; thus, for example, the simple equation (9) is replaced by

$$h_t = -\left[\frac{h^3}{3\mu}(\rho g + \sigma h_{sss})\right]_s - E(s,t). \tag{17}$$

Equations (14) – (17) form a complete system, to be solved numerically, that incorporates gravity drainage, evaporation, and drying.

It may be that other physical effects need to be incorporated. Often, when a coating compound dries, the surface tension changes. This is the case for alkyd paints, for example. If surface tension is a function of concentration, surface tractions will develop that tend to drive the coating to regions where σ is greater, as discussed in Landau and Lifshitz (1959). More complex rheological laws are needed if the viscosity is observed to be a function of flow rate. Surfactants may also be present in the coating compound and their diffusion and transport will affect the flow. Modeling studies including both surface-tension-gradient and complex rheology for liquid coatings may be found in Schwartz and Eley (1994) and Weidner *et al.* (1994).

5. Conclusions

The experimental results presented to the MISG reported three major defects in the final dry films, for drawn coatings. These were (i) long wavelength variation in thickness, leading to evenly-spaced interference fringes, (ii) a thick edge or lip at the bottom of coated lens blanks, and (iii) periodic undulations in coating thickness, with wavelengths of about 2 mm. Measured average, or central, thicknesses were reported as a function of drawing speed, resin concentration, and initial viscosity. For low-speed drawing, the Landau-Levich theory, discussed in section 2, provides good estimates of thicknesses, as well as providing an explanation for long wavelength variation in thickness. At the higher speeds, drainage has been shown to lead to uniform thickness gradients with thicker coating near the bottom of the lens. In all cases, drainage produces pronounced thick edges on the bottom. Simulation results reported here do not, as yet, provide an explanation for short wavelength undulations. It is possible that they arise during drying and that the improved model, outlined in section 4, will reproduce them. We note that the extent of the lower fat edge, which scales as $L_C^{2/3}h_0^{1/3}$, is in the neighbourhood of 2 mm, and that, immediately above the fat edge, the coating is relatively thin. This thin region may be expected to dry relatively rapidly and become less mobile. Liquid may then 'pile-up' behind it, producing another fat edge, and a thin region above it. This recurring process, during drainage, may lead to the reported periodic undulations.

Clearly the most uniform coatings are produced at slow withdrawal rates, when drainage is less important. Variations in thickness due to lens curvature could be mitigated by varying the drawing speed, in accordance with the varying inclination angle, as the lens is pulled from the bath. This may not constitute a practical solution for several reasons, however; very low speeds will produce coatings that may be too thin, and variable drawing speed would not seem to be consistent with large-batch processing.

Tailoring the properties of the coating to the application is perhaps a more promising approach. By implementing a more complicated numerical model, as in section 4, suitable combinations of solids fraction, viscosity, and drying rate may be established. Model development will also require more experimental data. It is especially important to obtain detailed measurements of final coating profiles, information concerning drying rates and viscosity variation with solids fraction. Considering the scale of SOLA's business and the economic benefit from simple, rapid coating operations, additional research on this problem would seem to be warranted.

Acknowledgements

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